# A zwitterionic zirconocene alkyl complex as a single-component alpha-olefin dimerization catalyst

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## **Supporting Material** to:

"A Zwitterionic Zirconocene Alkyl Complex as a Single-component $\alpha$-Olefin Dimerization Catalyst"

by H. van der Heijden, B. Hessen, and A. G. Orpen

*Part I: Experimental Section*

### General

All experiments were performed under nitrogen atmosphere using standard glove-box and Schlenk techniques. Deuterated solvents (Aldrich, Acros) were flushed with nitrogen, dried over molecular sieves and stored and used in a glove-box. Bromobenzene was distilled from $CaH_2$ under nitrogen and stored over mol. sieves. Other solvents were either flushed with nitrogen and dried over sodium wire, or distilled under argon atmosphere from sodium or sodium benzophenone ketyl. Commercially available $Cp^*ZrCl_3$ (Strem) was used as purchased. $B(C_6F_5)_3$ was obtained in hydrocarbon solution (AKZO Nobel) and recovered as a solid after removal of the solvent *in vacuo*. 1-Pentene (Aldrich) was degassed, stored over mol. sieves and vacuum transferred before use. Propene (Scott, 99.5%) was passed over BASF R 3-11 scavenger and mol. sieves before being fed into the reactor. NMR spectra were run on Varian XL-200, Gemini 300 and Inova 400 spectrometers.

### $Cp^*ZrNp_2Cl$

This is a modification of the procedure described in: Wengrovius, J. H.; Schrock, R. R. *J. Organomet. Chem.* **1981**, *205*, 319.

Solid $Np_2Mg$.dioxane (1.515 g, 5.95 mmol) was added to a suspension of $Cp^*ZrCl_3$ (1.98 g, 5.59 mmol) in 50 mL of diethyl ether. The resulting mixture was stirred at ambient temperature for 2 days, after which the solids were removed by centrifugation. From the solution the solvent was removed *in vacuo* to give a white solid. This was taken up in pentane and residual solids were removed by centrifugation. Evaporation of the solvent gave a white solid. Recrystallization from 2 mL of pentane at -40°C yielded 1.05 g (2.6 mmol, 44%) of $Cp^*ZrNp_2Cl$ as a white crystalline solid. Removal of the solvent from the mother liquor *in vacuo* yielded another 1.0 g (2.5 mmol, 42%) of white solid $Cp^*ZrNp_2Cl$.

$^1$H-NMR ($C_6D_6$, 300 MHz, 25°C): δ 1.97 (s, Cp*), 1.35 (d, $J$ = 13.0 Hz, ZrC*H*H), 1.05 (s, C*Me*$_3$), 0.26 (d, $J$ = 13.0 Hz, ZrCH*H*).

**Cp*(*t*-BuC$_5$H$_4$)ZrNp$_2$**

A solution of *t*-BuC$_5$H$_4$Li (0.480 g, 3.75 mmol) in 3 mL of THF was added to a solution of $Cp^*ZrNp_2Cl$ (1.40 g, 3.46 mmol) in 6 mL of THF. The resulting mixture was stirred at ambient temperature for 1 h, after which the solvent was removed *in vacuo*. The residue was stirred up with some pentane, which was subsequently pumped off (to remove residual THF). Again, pentane was added to the residue, and solids were removed by centrifugation. Removal of the solvent yielded crude Cp*(*t*-BuC$_5$H$_4$)ZrNp$_2$ (1.40 g, 2.86 mmol, 82%) as a yellow solid. Recrystallization from 2 mL of pentane at -40°C produced 0.78 g (1.59 mmol, 46%) of pure Cp*(*t*-BuC$_5$H$_4$)ZrNp$_2$.

$^1$H-NMR ($C_6D_6$, 300 MHz, 25°C): δ 6.22, 5.75 (m, Cp C*H*), 1.78 (s, Cp*), 1.23 (s, CpC*Me*$_3$), 1.21 (s, C*Me*$_3$), 1.09 (d, $J$ = 11.8 Hz, ZrC*H*H), -0.53 (d, $J$ = 11.8 Hz, ZrCH*H*).

$^{13}$C-NMR ($C_6D_6$, 75.4 MHz, 25°C): δ 144.4 (s, Cp *C*), 117.8 (s, Cp* *C*), 107.5 (d, $J$ = 170 Hz, Cp *C*H), 106.2 (d, $J$ = 168 Hz, Cp *C*H), 73.3 (t, $J$ = 100 Hz, Zr*C*H$_2$), 36.5 (s, CH$_2$*C*Me$_3$), 35.4 (q, $J$ = 125 Hz, CH$_2$C*Me*$_3$), 33.8 (s, Cp*C*Me$_3$), 32.1 (q, $J$ = 125 Hz, CpC*Me*$_3$), 12.8 (q, $J$ = 126 Hz, Cp**Me*).

Anal. Calcd for $C_{29}H_{50}Zr$ (%): C, 71.10; H, 10.29. Found: C, 70.94; H, 10.19.

**Cp*(C$_5$H$_4$CMe$_2$CH$_2$)ZrNp (1)**

A solution of Cp*(*t*-BuC$_5$H$_4$)ZrNp$_2$ (0.710 g, 1.45 mmol) in 40 mL of benzene was heated at 80°C for 16 h, after which the solvent was removed *in vacuo*. Remaining volatiles were removed by dissolution in pentane which was subsequently pumped off. The residue was dissolved in pentane and centrifuged (to remove traces of precipitate). Removal of the solvent *in vacuo* produced 0.505 g (1.20 mmol, 83%) of crude **1** as a yellow solid. Recrystallization from 1 mL of pentane at -40°C yielded 0.265 g (0.63 mmol, 44%) of pure **1**.

$^1$H-NMR ($C_6D_6$, 300 MHz, 25°C): δ 6.76, 6,46, 4.88, 4.78 (m, Cp C*H*), 1.74 (s, Cp*), 1.54 (s, Me), 1.18 (s, Me), 1.17 (d, *J* = 11.7 Hz, Np ZrC*H*H), -0.54 (d, *J* = 11.6 Hz, *exo*-ZrC*H*H), -0.69 (d, *J* = 11.7 Hz, Np ZrCH*H*), -2.09 (d, *J* = 11.6 Hz, *endo*-ZrCH*H*).
$^{13}$C{$^1$H}-NMR ($C_6D_6$, 75.4 MHz, 25°C): δ 119.4 (Cp *C*), 116.9 (Cp* *C*), 112.6, 107.1, 106.9, 104.2 (Cp *C*H), 74.8 (Zr*C*$H_2$CMe$_3$), 37.8 (Zr*C*$H_2$), 36.5 ($CH_2$*C*Me$_3$), 35.8 ($CH_2$C*Me*$_3$), 33.9 (*C*Me$_2$), 31.3 (Me), 29.1 (Me), 12.0 (Cp**Me*).
Anal. Calcd for $C_{24}H_{38}Zr$ (%): C, 69.00; H, 9.17. Found: C, 68.74; H, 9.04.

**Cp*[$C_5H_4CMe_2CH_2B(C_6F_5)_3$]ZrNp (2)**

A mixture of **1** (40 mg, 0.095 mmol) and $B(C_6F_5)_3$ (51.2 mg, 0.10 mmol) was dissolved in 1 mL of toluene at ambient temperature. After standing for 5 min the solution was cooled to -40°C, precipitating a deep yellow oil. The supernatant was decanted and the oil rinsed with pentane and dried *in vacuo* (repeated twice). This produced 50 mg of **2** as a yellow solid. By NMR, the solid contains 0.5 equiv.of toluene per formula unit. Yield: 0.050 mmol of **2**. 0.5 ($C_7H_8$), 53%. The compound is difficult to crystallize, but cooling concentrated solutions of **2** in 1,1,2,2-tetrachloroethane to -40°C produces (poly)crystalline material.

$^1$H-NMR ($C_6D_5Br$, 300 MHz, 25°C): δ 6.85, 6.75, 5.69, 4.89 (m, Cp C*H*), 4.29 (d, *J* = 7.0 Hz, ZrC*H*H), 1.65 (s, Cp*), 1.32 (s, Me), 1.16 (s, C*Me*$_3$), 0.75 (br, BC*H*H), 0.44 (s, Me), -0.15 (br, BCH*H*), -1.13 (d, *J* = 7.0 Hz, ZrCH*H*).
$^{13}$C-NMR ($C_2D_2Cl_4$, 100.6 MHz, -20°C): δ 148.8 (br d, *o*-*C*F), 141.21 (s, Cp *C*), 138.5 (br d, *p*-*C*F), 137.2 (br d, *m*-*C*F), 129.80 (dd, *J* = 101, 111 Hz, Zr*C*$H_2$), 125.02 (s, Cp* *C*), 123.8 (br s, $C_6F_5$ *C*), 114.78 (d, *J* =178 Hz, Cp *C*H), 111.35 (d, *J* = 179 Hz, Cp *C*H), 110.43 (d, *J* = 143 Hz, Cp *C*H), 107.41 (d, *J* = 178 Hz, Cp *C*H), 43.25 (s, *C*Me$_3$), 36.51 (s, *C*Me$_2$), 34.11 (q, *J* = 125 Hz, C*Me*$_3$), 28.13 (q, *J* = 128 Hz, Me), 25.21 (q, *J* = 126 Hz, Me), 13.0 (very br, B*C*$H_2$), 12.69 (q, *J* = 126 Hz, Cp**Me*).
$^{19}$F-NMR ($C_6D_5Br$, 282 MHz, 25°C): δ -130.5 (br, *o*-F), -160.8 (br t, *p*-F), 165.3 (br, *m*-F).

$^{19}$F-NMR ($C_6D_5Br$, 282 MHz, -40°C): δ -126.9 (2x), -129.5 (2x), -131.2, -132.2 (*o*-F), -158.3, -158.5, -158.9 (*p*-F), -161.0, -162.1, -163.1, -163.4, -163.7, -165.0 (*m*-F). The compound was thermally too unstable to send out for elemental analysis.

**Thermal decomposition of 2**

A solution of **2** in $C_6D_5Br$ was allowed to stand at ambient temperature (20°C). By NMR gradual disappearance of **2** with a half-life time of about 1 h was observed, accompanied by formation of Cp*(*t*-$BuC_5H_4$)Zr($\eta^3$-2-$C_4H_7$)[MeB($C_6F_5$)$_3$ (**3**). No intermediates in the reaction (including isobutene) could be observed.

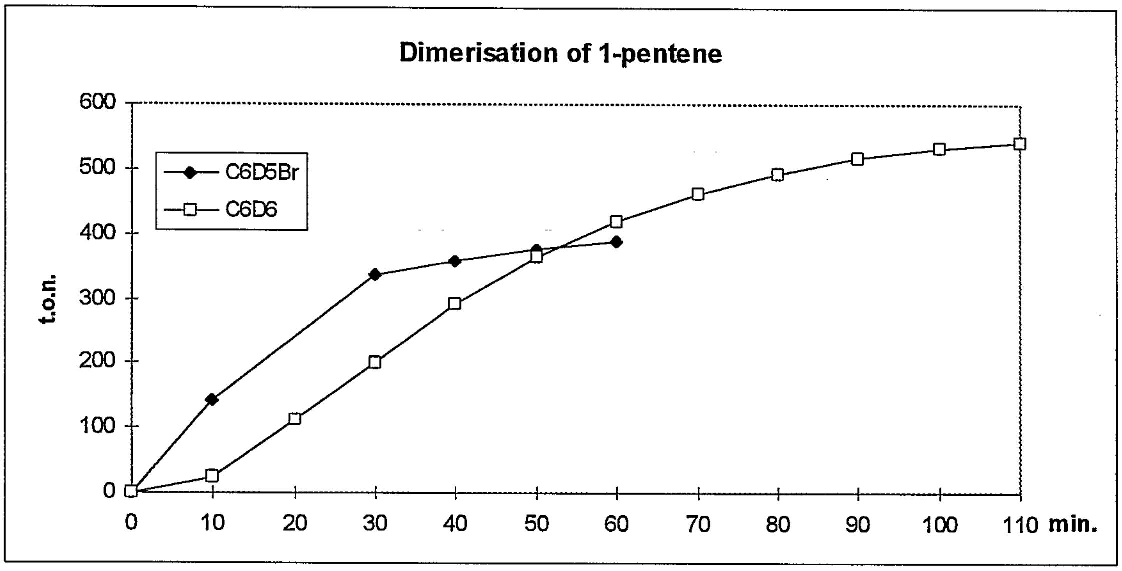
$^1$H-NMR ($C_6D_5Br$, 300 MHz, 25°C): δ 6.14, 5.94, 5.67, 4.68 (m, CpC*H*), 3.54, 2.92, 2.58, 1.91 (m, allyl CH*H*), 2.04 (s, Me), 1.60 (s, Cp*), 1.13 (br, BC*H*$_3$), 0.36 (s, C*Me*$_3$). $^{19}$F-NMR ($C_6D_5Br$, 188 MHz, 25°C): δ -133.15 (*o*-F), -165.31 (*p*-F), -167.84 (*m*-F). $^{13}$C-NMR ($C_6D_5Br$, 75.4 MHz, 25°C): δ 170.2 (s, allyl *C*), 148.8 (d, $J_{CF}$ = 135 Hz, *o*-*C*F), 142.4 (s, Cp *C*), 137.7 (d, $J_{CF} \approx$ 140 Hz, *p*-*C*F), 136.8 (d, $J_{CF} \approx$ 140 Hz, *m*-*C*F), 124.4 (s, Cp* *C*), 117.0 (d, *J* = 174 Hz, Cp *C*H), 113.0 (d, *J* = 174 Hz, Cp *C*H), 108.9 (d, *J* = 176 Hz, Cp *C*H), 106.9 (d, *J* = 173 Hz, Cp *C*H), 78.7 (t, *J* = 148 Hz, *C*H$_2$), 31.5 (s, *C*Me$_3$), 30.7 (q, $J \approx$ 124 Hz, allyl Me), 24.1 (q, *J* = 125 Hz, C*Me*$_3$), 12.3 (very br, BCH$_3$), 12.1 (q, *J* = 125 Hz, Cp**Me*).

**Catalytic dimerization of propene with 2**

A 0.5 l stainless steel (Büchi) autoclave was charged with 150 mL of toluene and thermostated at 25°C after which it was pressurized with 3.5 bar of propene. A solution of 45 mg (0.107 mmol) of **1** and 60 mg (0.117 mmol) of B($C_6F_5$)$_3$ in 5 mL of toluene was injected and the mixture stirred under constant propene pressure for 1 h. The mixture was quenched with methanol and analyzed by GC (using added benzene as standard). The product consisted of 2-methyl-1-pentene (36.0 mmol, corresponding to 336 turnovers per Zr) and less than 1% of trimer ($C_9$).

**Catalytic dimerization of 1-pentene with 2**

A solution of **2** in $C_6D_6$ was made by dissolving 3.5 mg (8.4 μmol) of **1** and 4.5 mg (8.8 μmol) of B($C_6F_5$)$_3$ in 0.4 mL of $C_6D_6$. Subsequently 440 mg (6.3 mmol) of 1-pentene was added and the conversion monitored at room temperature by NMR over a period of 110 min. (see figure below, showing total amount of 1-pentene turned over as function of time). By NMR and GC the product was analyzed as 2-propyl-1-heptene (>99%). A similar experiment in $C_6D_5Br$ solvent showed faster initial conversion of 1-pentene, but also a more rapid deactivation (see figure).




Analysis of the solution containing the deactivated catalyst by negative ion Electrospray MS (using wet acetonitrile carrier solvent) showed no presence of $C_5$- or $C_{10}$-alkyl$B(C_6F_5)_3$-anions (which could be indicative of ligand-to-alkyl borane transfer), but only a species with a mass (633) consistent with a formulation as the $C_5H_5CMe_2CH_2B(C_6F_5)_3$-anion. This species is expected to be produced on hydrolysis of a species containing the $[C_5H_4CMe_2CH_2B(C_6F_5)_3]^{2-}$ ligand.

**Oligomerization of 1-pentene with various reference compounds**

On NMR-tube scale ($C_6D_5Br$ solvent) experiments were carried out using 175 mg 1-pentene (2.5 mmol) and 10 μmol of various catalysts/activator combinations to estimate relative oligomer distributions. Oligomer ratios were estimated by GC integrated intensity divided by the number of C-atoms in the product.

**1** + $B(C_6F_5)_3$ : $C_{10}$ (100), $C_{15}$ (0.5), higher oligomers not observed.

Cp*(*t*-BuCp)$ZrMe_2$ + $B(C_6F_5)_3$ : $C_{10}$ (100), $C_{15}$ (46), $C_{20}$ (20), $C_{25-30}^{+}$ observed.

Cp*(*t*-BuCp)$ZrMe_2$ + $[Ph_3C][B(C_6F_5)_4]$ : $C_{10}$ (100), $C_{15}$ (79), $C_{20}$ (46), $C_{25}$ (35), $C_{30}^{+}$ observed.

**1** + $[Ph_3C][B(C_6F_5)_4]$ : $C_{10}$ (100), $C_{15}$ (72), $C_{20}$ (33), $C_{25-30}^{+}$ observed.

**$Cp^*[C_5H_4CMe_2CH_2B(C_6F_5)_3]Zr(\eta^3\text{-}2\text{-}C_4H_7)$ (4)**

In a Schlenk tube equipped with a rubber septum, a solution of $Cp^*[\eta^5\text{-}C_5H_4CMe_2CH_2B(C_6F_5)_3]ZrNp(toluene)_{0.5}$ (115 mg, 0.118 mmol) in 7 mL of bromobenzene was cooled to -50$^0$C. By gas-tight syringe, 50 mL of gaseous isobutene and 6 mL of $H_2$ were added. The resulting mixture was stirred for 15 min. during which it was allowed to warm to room temperature. The solvent was subsequently removed in vacuo and the solid residue stripped of remaining solvent by adding and then pumping off 5 mL of pentane. Washing with pentane yielded $Cp^*[CpCMe_2CH_2B(C_6F_5)_3]Zr(\eta^3\text{-}2\text{-}MeC_3H_4)$ as a yellow solid, which contained some residual bromobenzene (NMR). The yield was not determined, but a similar experiment performed in an NMR tube showed that conversion to **4** is essentially quantitative. Crystals suitable for X-ray diffraction were obtained by dissolving the material obtained above in a few mL of $CH_2Cl_2$ and cooling the solution to -40$^0$C. The resultant yellow crystals contained 1.5 eq. of $CH_2Cl_2$ per molecule of **4** (NMR).

$^1$H-NMR ($CD_2Cl_2$, 400 MHz, 25°C): $\delta$ 6.53, 6.02, 5.94, 4.99 (m, 1H each, Cp CH), 3.85 (d, $J$ = 1.2 Hz, 1H, =$CH_2$), 3.51 (dd, $J$ = 1.2, 3.5 Hz, 1H, =$CH_2$), 3.02 (d, $J$ = 3.7 Hz, 1H, =$CH_2$), 2.45 (s, 3H, allyl-Me), 2.37 (dd, $J$ = 3.5, 3.7 Hz, 1H, =$CH_2$), 1.97 (s, 15H, Cp*), 1.04 (br d, $J$ = 14 Hz, $CH_2B$), 0.79 (br d, $J$ = 14 Hz, $CH_2B$), 0.62 (s, 3H, Me), 0.49 (s, 3H, Me).

$^{13}$C{$^1$H}-NMR ($CD_2Cl_2$, 100.6 MHz, 25°C): $\delta$ 166.6 (*ipso*-allyl), 148.5 (o-$C_6F_5$), 146.8 (Cp *ipso* C), 138.3 (p-$C_6F_5$), 136.6 (m-$C_6F_5$), 126.5 (i-$C_6F_5$), 123.50 ($Me_5\mathit{C_5}$), 117.8, 110.4, 107.1, 105.9 (all Cp CH), 79.6, 63.3 (both =$CH_2$), 36.3 ($Me_2\mathit{C}$), 30.6 (allyl Me), 25.2, 21.1 (both Me), 24 (br, $CH_2B$), 12.6 ($\mathit{Me_5}C_5$).

$^{19}$F-NMR ($CD_2Cl_2$, 282 MHz, 25°C): $\delta$ -130.0 (6F, *o*-F), -164.4 (3F, *p*-F), -166.7 (6F, *m*-F).

**Supporting Material** to:

"A Zwitterionic Zirconocene Alkyl Complex as a Single-component $\alpha$-Olefin Dimerization Catalyst"

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*Part II: Crystal Structure Determination*

of $(C_5Me_5)[C_5H_4CMe_2CH_2B(C_6F_5)_3]Zr(\eta^3\text{-}CH_2CMeCH_2)$

Structure Analysis of $[ZrCp^*\{\eta^5\text{-}C_5H_4CMe_2CH_2B(C_6F_5)_3\}(\eta^3\text{-}CH_2CMeCH_2)].1.5CH_2Cl_2$ (**1**)$.1.5CH_2Cl_2$

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## Experimental

Crystal data and other details of the structure analysis are presented in Table 1. A crystal of **1** was coated in oil and mounted on a glass fibre. All diffraction measurements were made at -100°C on a Siemens SMART CCD area detector diffractometer, using graphite monochromated Mo-$K_\alpha$ *X*-radiation. Unit cell dimensions were determined from the positions of 198 reflections in 90 intensity frames measured at 0.3° intervals in $\omega$. Diffracted intensities were measured in a hemisphere of reciprocal space for $2\theta<46.5°$ by 0.3° width $\omega$ frames, a total of 1321 frames being measured in 14 hours. No decay over the period of data collection was observed. A total of 16760 integrated intensity data were collected, rocking curves were notably broad (full width > 2° in many cases). Of the 11634 unique observations remaining after averaging of duplicate and equivalent measurements and deletion of systematic absences, 11256 with $I>-1.5\sigma(I)$ were retained for use in structure refinement. An absorption correction was applied on the basis of 3124 equivalent reflection intensities, transmission coefficients were in the range 0.849 to 0.731. Lorentz and polarisation corrections were applied.

The structure was solved by Patterson and Fourier methods. All non-hydrogen atoms were assigned anisotropic displacement parameters and refined without positional constraints. All hydrogen atoms were constrained to idealised geometries (except those of the methylene groups of the allyl ligands which were refined without constraints) and assigned isotropic displacement parameters based on those of their attached carbon atoms. Full-matrix least-squares refinement of this model against $F^2$ converged to final residual indices given in Table 1. Weights, w, were set equal to $[\sigma_c^2(F_o^2)+(aP)^2 + bP]^{-1}$, where $P = [0.333\max\{F_o^2,0\} + 0.667F_c^2]$, $\sigma_c^2(F_o^2)$ is the variance in $F_o^2$ due to counting statistics, and a = 0.015, b = 35 were chosen to minimise the variation in S as a function of $|F_o|$. Final difference electron density maps showed no features of chemical significance with all peaks of magnitude > 0.5 eÅ$^{-3}$ lying close to the solvent molecules. These peaks and the highly anisotropic displacement parameters of the solvent atoms and the distorted geometries of these molecules imply that the dichloromethane molecules are disordered. However no satisfactory multi-site model was refined. Table 2 lists the final atomic positional parameters for the non-hydrogen atoms, Table 3 the derived bond lengths and inter-bond angles, and Tables 4-6 the anisotropic displacement parameters, hydrogen atomic parameters and observed and calculated structure factors respectively. All calculations were carried out using programs of the SHELXTL package.[1] Complex neutral-atom scattering factors were taken from reference 2.

## Discussion

Perspective views of the molecular structure of (**1**) are given in Figures 1-8. The crystal structure consists of isolated molecules of **1** and dichloromethane separated by normal van der Waals contacts. There are two independent molecules of **1** and three of dichloromethane in the asymmetric unit Molecules of **1** consist of a zirconium(IV) centre coordinated by an $\eta^5$-$C_5Me_5$ (Cp*) ligand, a second cyclopentadienyl ligand $\{\eta^5$-$C_5H_4CMe_2CH_2B(C_6F_5)_3\}$ (cp-BPf3) and an $\eta^3$-2-methallyl ligand. The complex structure is of the usual bent metallocene type but with some asymmetry in the binding of the allyl ligand (presumably as a consequence of the asymmetry in bulk of the cp-BPf3 ligand).

## References

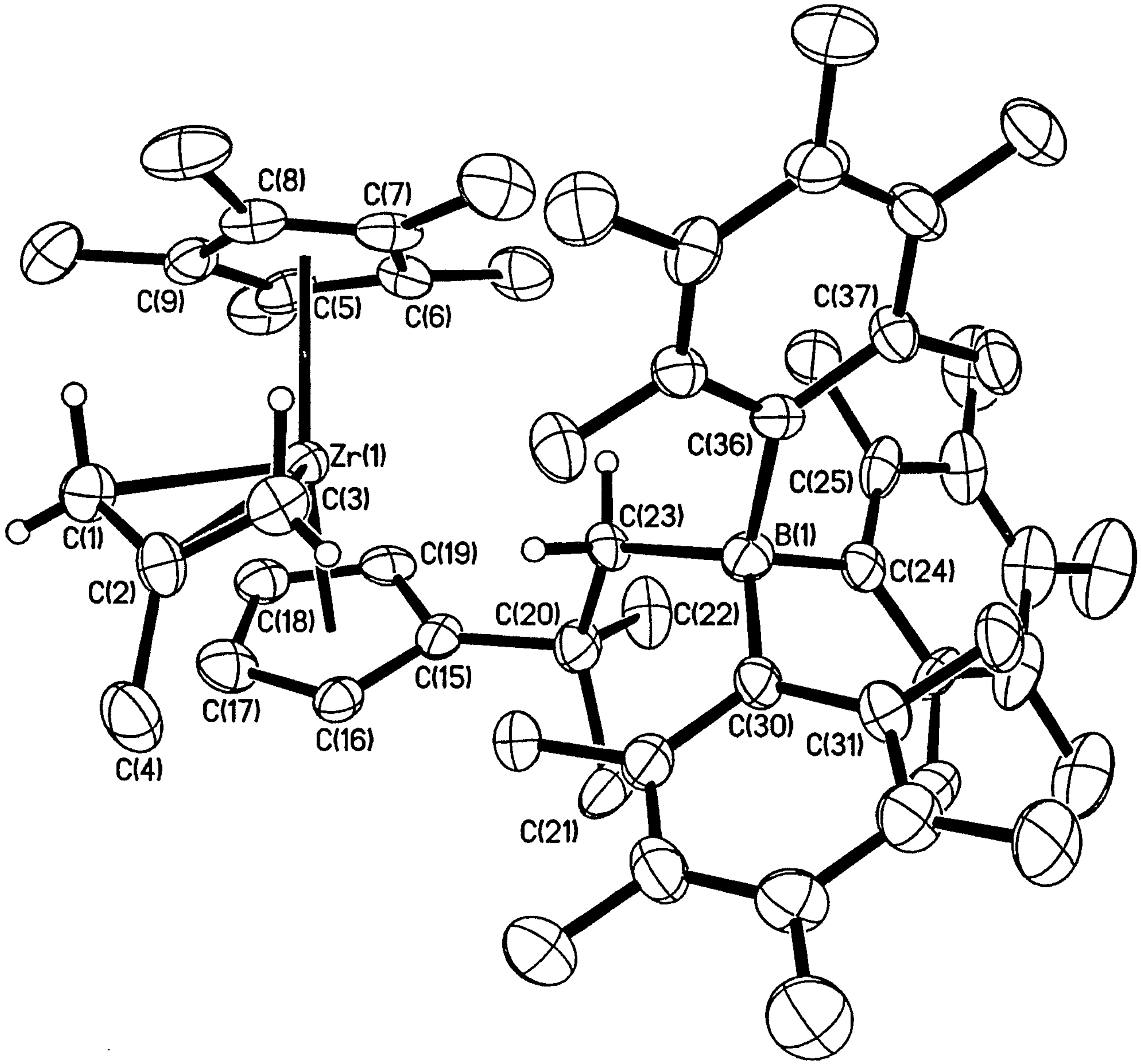

1. SHELXTL Rev. 5.0, Siemens Analytical X-ray, 1994.
2. *International Tables for Crystallography*, Vol. C, 1992, Kluwer, Dordrecht.


## Captions for Figures

Figure 1. Molecular structure of the first independent molecule of **1** showing atom labelling scheme. All methyl group hydrogen atoms have been omitted for clarity.

Figure 2. Molecular structure of the second independent molecule of **1** showing atom labelling scheme. All methyl group hydrogen atoms have been omitted for clarity.

Figure 3. Molecular geometry of the first independent molecule of **1** showing atom labelling scheme. Non-hydrogen atoms are represented as ellipsoids enclosing 50% probability density. All methyl group hydrogen atoms have been omitted for clarity.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C(9)
C(5)
C(6)
C(37)
C(36)
Zr(1)
C(3)
C(25)
C(23)
B(1)
C(1)
C(19)
C(24)
C(2)
C(20)
C(22)
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C(16)
C(30)
C(31)
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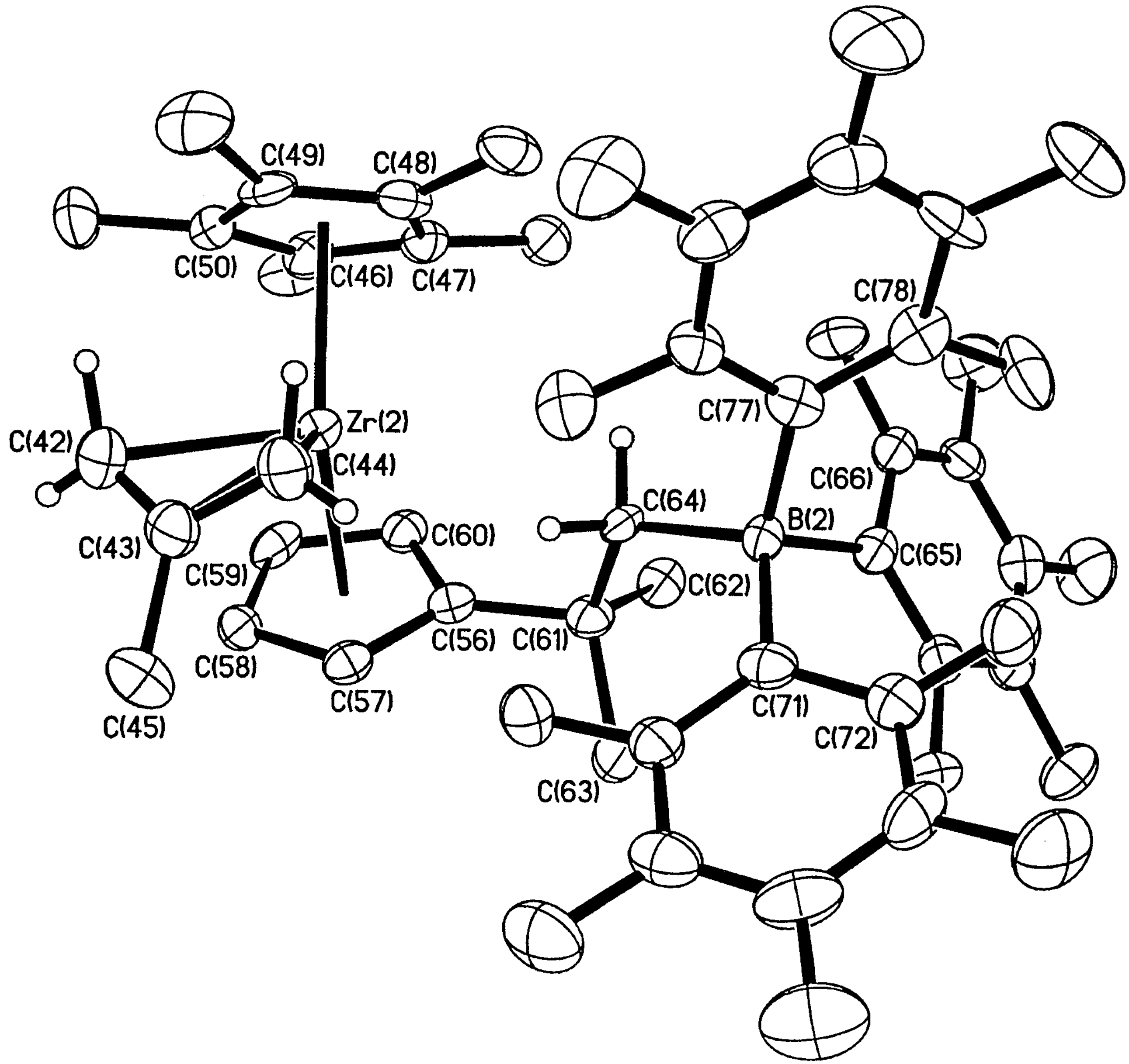
C(21)



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Zr(2)
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C(59)
C(62)
C(56)
C(61)
C(58)
C(57)
C(71)
C(72)
C(45)
C(63)

| | |
|---|---|
| Identification code | cqtrezr |
| Empirical formula | $C_{42.5}$ $H_{37}BCl_3F_{15}Zr$ |
| Formula weight | 1041.10 |
| Temperature | 173 K |
| Wavelength | 0.71073 Å |
| Crystal system | triclinic |
| Space group | P-1 (No. 2) |
| Unit cell dimensions | a = 12.344(2) Å  $\alpha$ = 107.50(2) °<br>b = 18.086(3) Å  $\beta$ = 92.770(12) °<br>c = 20.101(3) Å  $\gamma$ = 94.766(10)° |
| Volume | 4252.6(12) $Å^3$ |
| Z | 4 |
| Density (calculated) | 1.626 $Mg/m^3$ |
| Absorption coefficient | 0.545 $mm^{-1}$ |
| F(000) | 2092 |
| Crystal size | .12 x .25 x .25 mm |
| Theta range for data collection | 2.00 to 23.25 ° |
| Index ranges | -14<=h<=14, -21<=k<=21, -14<=l<=23 |
| Reflections collected | 16760 |
| Independent reflections | 11634 [R(int) = 0.0410] |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 11256 / 0 / 1166 |
| Goodness-of-fit on $F^2$ (S)* | 1.263 |
| Final R indices* [$I>2\sigma(I)$] | R1 = 0.0814, wR2 = 0.1557 [8727 data] |
| R indices* (all data) | R1 = 0.1128, wR2 = 0.1769 [11256 data] |
| Largest diff. peak and hole | 1.46 and -1.25 $e.Å^{-3}$ |

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*$wR2 = [\sum w\Delta^2 / \sum wF_o^4]^{0.5}$ ; $S = [\sum w\Delta^2 / (N.O. - N.V.)]^{0.5}$ ; $\Delta = F_o^2 - F_c^2$ ;
$R1 = \sum |F_o - F_c| / \sum |F_o|$ ; N.O. = data + restraints; N.V. = parameters

Table 2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$) for **1**. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Zr(1) | 2852(1) | 8991(1) | 6387(1) | 25(1) |
| Zr(2) | 7863(1) | 6563(1) | 9089(1) | 24(1) |
| Cl(1) | 2247(3) | 1300(3) | 4287(2) | 104(1) |
| Cl(2) | 2302(4) | 1608(3) | 2971(3) | 134(2) |
| Cl(3) | 8504(5) | 6992(3) | 4071(5) | 205(4) |
| Cl(4) | 7176(5) | 5596(5) | 3803(6) | 267(5) |
| Cl(5) | 7707(5) | 652(3) | -1893(3) | 149(2) |
| Cl(6) | 9601(5) | 626(4) | -917(2) | 154(2) |
| F(1) | 993(4) | 6323(3) | 4411(3) | 38(1) |
| F(2) | 306(5) | 4853(4) | 4277(3) | 64(2) |
| F(3) | 1699(6) | 3721(3) | 3925(3) | 75(2) |
| F(4) | 3794(6) | 4121(3) | 3690(3) | 60(2) |
| F(5) | 4498(4) | 5564(3) | 3792(2) | 38(1) |
| F(6) | 3657(4) | 6167(3) | 2779(2) | 38(1) |
| F(7) | 5401(5) | 6421(4) | 2123(3) | 55(2) |
| F(8) | 7028(5) | 7563(4) | 2791(3) | 56(2) |
| F(9) | 6818(4) | 8438(3) | 4134(3) | 47(1) |
| F(10) | 5098(4) | 8228(3) | 4788(2) | 37(1) |
| F(11) | 1286(4) | 6063(3) | 3029(2) | 36(1) |
| F(12) | -5(4) | 6686(3) | 2329(3) | 51(2) |
| F(13) | 181(5) | 8246(3) | 2520(3) | 57(2) |
| F(14) | 1735(4) | 9186(3) | 3492(3) | 45(1) |
| F(15) | 3034(4) | 8585(3) | 4235(2) | 36(1) |
| F(16) | 3496(4) | 5735(3) | 9355(2) | 32(1) |
| F(17) | 2029(4) | 4633(3) | 8560(3) | 44(1) |
| F(18) | 725(4) | 4920(3) | 7540(3) | 49(2) |
| F(19) | 959(4) | 6353(3) | 7352(3) | 43(1) |
| F(20) | 2414(4) | 7457(3) | 8127(2) | 30(1) |
| F(21) | 2204(4) | 8151(3) | 9511(3) | 39(1) |
| F(22) | 2005(5) | 9595(3) | 9515(3) | 53(2) |
| F(23) | 3710(6) | 10486(3) | 9244(3) | 63(2) |
| F(24) | 5673(5) | 9882(3) | 8998(3) | 50(2) |
| F(25) | 5914(4) | 8462(3) | 9011(3) | 37(1) |
| F(26) | 2150(4) | 6951(3) | 10074(3) | 43(1) |
| F(27) | 2124(5) | 7341(3) | 11433(3) | 54(2) |
| F(28) | 3954(5) | 8014(4) | 12288(3) | 56(2) |
| F(29) | 5836(5) | 8291(3) | 11694(3) | 55(2) |
| F(30) | 5872(4) | 7952(3) | 10330(2) | 36(1) |
| C(1) | 3685(8) | 10317(5) | 6825(5) | 33(2) |
| C(2) | 4278(7) | 10052(5) | 6233(5) | 32(2) |
| C(3) | 3729(8) | 9661(6) | 5591(5) | 33(2) |
| C(4) | 5508(7) | 10078(6) | 6311(5) | 45(3) |
| C(5) | 1098(7) | 8930(5) | 6979(4) | 31(2) |
| C(6) | 896(7) | 8430(5) | 6277(5) | 32(2) |
| C(7) | 932(6) | 8908(6) | 5837(4) | 30(2) |
| C(8) | 1164(7) | 9693(5) | 6259(5) | 34(2) |
| C(9) | 1284(7) | 9710(5) | 6968(4) | 30(2) |
| C(10) | 918(8) | 8704(6) | 7633(5) | 45(3) |
| C(11) | 442(7) | 7589(5) | 6074(5) | 42(2) |
| C(12) | 649(8) | 8635(7) | 5061(5) | 51(3) |
| C(13) | 1107(8) | 10388(6) | 6001(6) | 50(3) |
| C(14) | 1425(8) | 10423(6) | 7601(5) | 51(3) |
| C(15) | 3812(7) | 7796(5) | 6301(4) | 27(2) |
| C(16) | 4619(7) | 8437(5) | 6492(4) | 26(2) |
| C(17) | 4442(7) | 8913(6) | 7169(4) | 36(2) |
| C(18) | 3495(7) | 8590(5) | 7381(4) | 31(2) |
| C(19) | 3118(7) | 7904(5) | 6851(4) | 27(2) |
| C(20) | 3706(7) | 7135(5) | 5610(4) | 30(2) |
| C(21) | 4865(8) | 6908(6) | 5430(5) | 42(2) |
| C(22) | 3058(8) | 6425(5) | 5734(5) | 41(2) |
| C(23) | 3084(7) | 7430(5) | 5048(4) | 23(2) |
| C(24) | 2772(7) | 6037(5) | 4082(4) | 24(2) |
| C(25) | 1745(8) | 5789(5) | 4222(4) | 31(2) |

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|---|---|---|---|---|
| C(27) | 2031(10) | 4462(6) | 3989(5) | 48(3) |
| C(28) | 3083(10) | 4667(5) | 3865(5) | 44(3) |
| C(29) | 3440(7) | 5431(5) | 3922(4) | 28(2) |
| C(30) | 4292(7) | 7144(5) | 3839(4) | 27(2) |
| C(31) | 4441(7) | 6738(5) | 3149(4) | 30(2) |
| C(32) | 5329(8) | 6849(6) | 2800(5) | 37(2) |
| C(33) | 6141(7) | 7431(6) | 3125(5) | 37(2) |
| C(34) | 6032(7) | 7863(5) | 3797(5) | 37(2) |
| C(35) | 5129(7) | 7725(5) | 4135(4) | 30(2) |
| C(36) | 2231(6) | 7290(5) | 3711(4) | 23(2) |
| C(37) | 1443(7) | 6846(5) | 3197(4) | 27(2) |
| C(38) | 755(7) | 7154(5) | 2807(5) | 35(2) |
| C(39) | 844(7) | 7935(5) | 2889(5) | 35(2) |
| C(40) | 1629(7) | 8402(5) | 3377(4) | 31(2) |
| C(41) | 2277(7) | 8080(5) | 3770(4) | 27(2) |
| C(42) | 9597(8) | 7366(6) | 9375(6) | 41(3) |
| C(43) | 8892(7) | 7951(5) | 9444(5) | 34(2) |
| C(44) | 8012(8) | 7955(5) | 9843(5) | 33(2) |
| C(45) | 9008(9) | 8507(6) | 9022(6) | 52(3) |
| C(46) | 8169(7) | 5236(5) | 9199(4) | 28(2) |
| C(47) | 7146(7) | 5422(5) | 9462(4) | 27(2) |
| C(48) | 7341(7) | 6060(5) | 10091(4) | 28(2) |
| C(49) | 8475(7) | 6261(5) | 10205(4) | 29(2) |
| C(50) | 8980(7) | 5779(5) | 9653(4) | 27(2) |
| C(51) | 8355(8) | 4526(5) | 8624(5) | 42(2) |
| C(52) | 6076(7) | 4921(5) | 9221(5) | 36(2) |
| C(53) | 6505(8) | 6359(5) | 10594(5) | 37(2) |
| C(54) | 9052(9) | 6825(6) | 10864(5) | 47(3) |
| C(55) | 10195(7) | 5713(6) | 9589(5) | 44(3) |
| C(56) | 6584(7) | 6423(5) | 8037(4) | 26(2) |
| C(57) | 7424(7) | 7017(5) | 8067(4) | 29(2) |
| C(58) | 8405(7) | 6667(6) | 7937(4) | 35(2) |
| C(59) | 8204(7) | 5885(5) | 7876(4) | 30(2) |
| C(60) | 7069(6) | 5729(5) | 7932(4) | 24(2) |
| C(61) | 5368(7) | 6529(5) | 8125(4) | 25(2) |
| C(62) | 4712(7) | 5729(5) | 7781(4) | 28(2) |
| C(63) | 5040(7) | 7090(5) | 7729(4) | 31(2) |
| C(64) | 5225(6) | 6851(5) | 8932(4) | 22(2) |
| C(65) | 3053(6) | 6659(5) | 8792(4) | 23(2) |
| C(66) | 2880(7) | 5919(5) | 8861(4) | 26(2) |
| C(67) | 2142(7) | 5337(5) | 8444(4) | 28(2) |
| C(68) | 1480(7) | 5470(5) | 7936(4) | 32(2) |
| C(69) | 1598(7) | 6201(5) | 7855(4) | 28(2) |
| C(70) | 2357(7) | 6767(5) | 8272(4) | 26(2) |
| C(71) | 4054(7) | 8189(5) | 9218(4) | 27(2) |
| C(72) | 3090(7) | 8549(5) | 9368(4) | 30(2) |
| C(73) | 2972(8) | 9300(5) | 9371(5) | 37(2) |
| C(74) | 3823(9) | 9762(5) | 9247(5) | 43(3) |
| C(75) | 4798(8) | 9451(5) | 9123(5) | 37(2) |
| C(76) | 4899(7) | 8683(5) | 9112(4) | 28(2) |
| C(77) | 4042(7) | 7409(5) | 10101(4) | 26(2) |
| C(78) | 3125(7) | 7274(5) | 10433(4) | 30(2) |
| C(79) | 3082(8) | 7474(5) | 11157(5) | 38(2) |
| C(80) | 3997(8) | 7818(5) | 11587(5) | 39(2) |
| C(81) | 4931(8) | 7962(5) | 11286(5) | 36(2) |
| C(82) | 4920(7) | 7761(5) | 10569(4) | 27(2) |
| C(83) | 1842(11) | 979(8) | 3408(7) | 81(4) |
| C(84) | 8360(16) | 6158(13) | 4162(15) | 204(13) |
| C(85) | 7979(15) | 678(10) | -1173(8) | 117(7) |
| B(1) | 3123(8) | 6980(6) | 4194(5) | 27(2) |
| B(2) | 4100(7) | 7271(5) | 9239(5) | 23(2) |

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|---|---|
| Zr(1)-C(1) | 2.412(10) |
| Zr(1)-C(18) | 2.442(8) |
| Zr(1)-C(19) | 2.455(8) |
| Zr(1)-C(17) | 2.495(9) |
| Zr(1)-C(16) | 2.501(8) |
| Zr(1)-C(3) | 2.513(9) |
| Zr(1)-C(6) | 2.514(9) |
| Zr(1)-C(15) | 2.515(8) |
| Zr(1)-C(5) | 2.532(8) |
| Zr(1)-C(7) | 2.540(8) |
| Zr(1)-C(9) | 2.555(8) |
| Zr(1)-C(8) | 2.570(8) |
| Zr(1)-C(2) | 2.593(8) |
| Zr(2)-C(42) | 2.426(10) |
| Zr(2)-C(59) | 2.453(8) |
| Zr(2)-C(60) | 2.464(8) |
| Zr(2)-C(57) | 2.485(8) |
| Zr(2)-C(58) | 2.496(8) |
| Zr(2)-C(44) | 2.503(9) |
| Zr(2)-C(47) | 2.509(8) |
| Zr(2)-C(56) | 2.513(8) |
| Zr(2)-C(50) | 2.517(8) |
| Zr(2)-C(46) | 2.533(9) |
| Zr(2)-C(48) | 2.535(8) |
| Zr(2)-C(49) | 2.558(8) |
| Zr(2)-C(43) | 2.594(9) |
| Cl(1)-C(83) | 1.721(13) |
| Cl(2)-C(83) | 1.710(14) |
| Cl(3)-C(84) | 1.57(2) |
| Cl(4)-C(84) | 1.70(2) |
| Cl(5)-C(85) | 1.46(2) |
| Cl(6)-C(85) | 2.06(2) |
| F(1)-C(25) | 1.379(10) |
| F(2)-C(26) | 1.374(11) |
| F(3)-C(27) | 1.334(11) |
| F(4)-C(28) | 1.353(11) |
| F(5)-C(29) | 1.360(10) |
| F(6)-C(31) | 1.364(9) |
| F(7)-C(32) | 1.358(10) |
| F(8)-C(33) | 1.353(10) |
| F(9)-C(34) | 1.352(10) |
| F(10)-C(35) | 1.358(9) |
| F(11)-C(37) | 1.349(9) |
| F(12)-C(38) | 1.346(10) |
| F(13)-C(39) | 1.343(10) |
| F(14)-C(40) | 1.360(9) |
| F(15)-C(41) | 1.356(9) |
| F(16)-C(66) | 1.357(9) |
| F(17)-C(67) | 1.359(9) |
| F(18)-C(68) | 1.338(10) |
| F(19)-C(69) | 1.357(9) |
| F(20)-C(70) | 1.361(9) |
| F(21)-C(72) | 1.349(10) |
| F(22)-C(73) | 1.357(10) |
| F(23)-C(74) | 1.331(10) |
| F(24)-C(75) | 1.357(10) |
| F(25)-C(76) | 1.353(10) |
| F(26)-C(78) | 1.363(10) |
| F(27)-C(79) | 1.364(10) |
| F(28)-C(80) | 1.350(10) |
| F(29)-C(81) | 1.334(10) |
| F(30)-C(82) | 1.347(9) |
| C(1)-C(2) | 1.408(12) |
| C(2)-C(3) | 1.382(13) |
| C(2)-C(4) | 1.514(12) |
| C(5)-C(9) | 1.417(12) |
| C(5)-C(6) | 1.426(12) |

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|---|---|
| C(6)-C(7) | 1.412(12) |
| C(6)-C(11) | 1.502(12) |
| C(7)-C(8) | 1.415(13) |
| C(7)-C(12) | 1.501(12) |
| C(8)-C(9) | 1.417(12) |
| C(8)-C(13) | 1.502(12) |
| C(9)-C(14) | 1.506(12) |
| C(15)-C(19) | 1.409(11) |
| C(15)-C(16) | 1.409(12) |
| C(15)-C(20) | 1.528(11) |
| C(16)-C(17) | 1.414(12) |
| C(17)-C(18) | 1.404(12) |
| C(18)-C(19) | 1.397(12) |
| C(20)-C(22) | 1.546(12) |
| C(20)-C(21) | 1.550(12) |
| C(20)-C(23) | 1.580(11) |
| C(23)-B(1) | 1.670(12) |
| C(24)-C(25) | 1.380(12) |
| C(24)-C(29) | 1.395(11) |
| C(24)-B(1) | 1.668(12) |
| C(25)-C(26) | 1.366(13) |
| C(26)-C(27) | 1.352(14) |
| C(27)-C(28) | 1.38(2) |
| C(28)-C(29) | 1.384(13) |
| C(30)-C(35) | 1.382(12) |
| C(30)-C(31) | 1.390(11) |
| C(30)-B(1) | 1.684(13) |
| C(31)-C(32) | 1.362(12) |
| C(32)-C(33) | 1.375(13) |
| C(33)-C(34) | 1.360(13) |
| C(34)-C(35) | 1.378(12) |
| C(36)-C(37) | 1.391(11) |
| C(36)-C(41) | 1.394(12) |
| C(36)-B(1) | 1.672(12) |
| C(37)-C(38) | 1.386(12) |
| C(38)-C(39) | 1.367(13) |
| C(39)-C(40) | 1.373(12) |
| C(40)-C(41) | 1.377(12) |
| C(42)-C(43) | 1.404(14) |
| C(43)-C(44) | 1.380(13) |
| C(43)-C(45) | 1.502(12) |
| C(46)-C(50) | 1.418(12) |
| C(46)-C(47) | 1.420(12) |
| C(46)-C(51) | 1.489(12) |
| C(47)-C(48) | 1.426(12) |
| C(47)-C(52) | 1.509(11) |
| C(48)-C(49) | 1.406(12) |
| C(48)-C(53) | 1.499(11) |
| C(49)-C(50) | 1.399(12) |
| C(49)-C(54) | 1.509(11) |
| C(50)-C(55) | 1.523(11) |
| C(56)-C(60) | 1.397(11) |
| C(56)-C(57) | 1.416(12) |
| C(56)-C(61) | 1.541(11) |
| C(57)-C(58) | 1.412(12) |
| C(58)-C(59) | 1.382(13) |
| C(59)-C(60) | 1.422(11) |
| C(61)-C(63) | 1.531(11) |
| C(61)-C(62) | 1.542(11) |
| C(61)-C(64) | 1.574(11) |
| C(64)-B(2) | 1.693(11) |
| C(65)-C(66) | 1.389(11) |
| C(65)-C(70) | 1.390(11) |
| C(65)-B(2) | 1.660(12) |
| C(66)-C(67) | 1.370(12) |
| C(67)-C(68) | 1.366(12) |
| C(68)-C(69) | 1.378(12) |
| C(69)-C(70) | 1.370(11) |

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|---|---|
| C(71)-C(76) | 1.385(12) |
| C(71)-C(72) | 1.406(12) |
| C(71)-B(2) | 1.679(12) |
| C(72)-C(73) | 1.375(12) |
| C(73)-C(74) | 1.366(14) |
| C(74)-C(75) | 1.372(14) |
| C(75)-C(76) | 1.400(12) |
| C(77)-C(82) | 1.376(12) |
| C(77)-C(78) | 1.381(12) |
| C(77)-B(2) | 1.680(12) |
| C(78)-C(79) | 1.395(12) |
| C(79)-C(80) | 1.370(13) |
| C(80)-C(81) | 1.369(13) |
| C(81)-C(82) | 1.376(12) |

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|---|---|
| C(1)-Zr(1)-C(18) | 96.2(3) |
| C(1)-Zr(1)-C(19) | 128.2(3) |
| C(18)-Zr(1)-C(19) | 33.1(3) |
| C(1)-Zr(1)-C(17) | 76.3(3) |
| C(18)-Zr(1)-C(17) | 33.0(3) |
| C(19)-Zr(1)-C(17) | 54.3(3) |
| C(1)-Zr(1)-C(16) | 93.0(3) |
| C(18)-Zr(1)-C(16) | 54.9(3) |
| C(19)-Zr(1)-C(16) | 54.4(3) |
| C(17)-Zr(1)-C(16) | 32.9(3) |
| C(1)-Zr(1)-C(3) | 58.6(3) |
| C(18)-Zr(1)-C(3) | 135.2(3) |
| C(19)-Zr(1)-C(3) | 139.5(3) |
| C(17)-Zr(1)-C(3) | 102.2(3) |
| C(16)-Zr(1)-C(3) | 87.5(3) |
| C(1)-Zr(1)-C(6) | 131.3(3) |
| C(18)-Zr(1)-C(6) | 98.4(3) |
| C(19)-Zr(1)-C(6) | 81.6(3) |
| C(17)-Zr(1)-C(6) | 131.2(3) |
| C(16)-Zr(1)-C(6) | 132.8(3) |
| C(3)-Zr(1)-C(6) | 126.3(3) |
| C(1)-Zr(1)-C(15) | 125.4(3) |
| C(18)-Zr(1)-C(15) | 54.9(3) |
| C(19)-Zr(1)-C(15) | 32.9(3) |
| C(17)-Zr(1)-C(15) | 54.2(3) |
| C(16)-Zr(1)-C(15) | 32.6(3) |
| C(3)-Zr(1)-C(15) | 107.1(3) |
| C(6)-Zr(1)-C(15) | 100.6(3) |
| C(1)-Zr(1)-C(5) | 107.1(3) |
| C(18)-Zr(1)-C(5) | 79.6(3) |
| C(19)-Zr(1)-C(5) | 80.0(3) |
| C(17)-Zr(1)-C(5) | 110.5(3) |
| C(16)-Zr(1)-C(5) | 132.3(3) |
| C(3)-Zr(1)-C(5) | 140.0(3) |
| C(6)-Zr(1)-C(5) | 32.8(3) |
| C(15)-Zr(1)-C(5) | 110.5(3) |
| C(1)-Zr(1)-C(7) | 112.0(3) |
| C(18)-Zr(1)-C(7) | 130.1(3) |
| C(19)-Zr(1)-C(7) | 112.5(3) |
| C(17)-Zr(1)-C(7) | 163.1(3) |
| C(16)-Zr(1)-C(7) | 152.0(3) |
| C(3)-Zr(1)-C(7) | 94.7(3) |
| C(6)-Zr(1)-C(7) | 32.4(3) |
| C(15)-Zr(1)-C(7) | 122.0(3) |
| C(5)-Zr(1)-C(7) | 53.5(3) |
| C(1)-Zr(1)-C(9) | 78.1(3) |
| C(18)-Zr(1)-C(9) | 96.7(3) |
| C(19)-Zr(1)-C(9) | 109.1(3) |
| C(17)-Zr(1)-C(9) | 117.1(3) |
| C(16)-Zr(1)-C(9) | 149.6(3) |
| C(3)-Zr(1)-C(9) | 111.2(3) |
| C(6)-Zr(1)-C(9) | 54.2(3) |
| C(15)-Zr(1)-C(9) | 141.6(3) |
| C(5)-Zr(1)-C(9) | 32.3(3) |

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|---|---|
| C(1)-Zr(1)-C(8) | 81.4(3) |
| C(18)-Zr(1)-C(8) | 128.5(3) |
| C(19)-Zr(1)-C(8) | 131.8(3) |
| C(17)-Zr(1)-C(8) | 146.3(3) |
| C(16)-Zr(1)-C(8) | 173.6(3) |
| C(3)-Zr(1)-C(8) | 86.9(3) |
| C(6)-Zr(1)-C(8) | 53.6(3) |
| C(15)-Zr(1)-C(8) | 153.2(3) |
| C(5)-Zr(1)-C(8) | 53.1(3) |
| C(7)-Zr(1)-C(8) | 32.1(3) |
| C(9)-Zr(1)-C(8) | 32.1(3) |
| C(1)-Zr(1)-C(2) | 32.4(3) |
| C(18)-Zr(1)-C(2) | 108.5(3) |
| C(19)-Zr(1)-C(2) | 129.5(3) |
| C(17)-Zr(1)-C(2) | 77.7(3) |
| C(16)-Zr(1)-C(2) | 77.1(3) |
| C(3)-Zr(1)-C(2) | 31.4(3) |
| C(6)-Zr(1)-C(2) | 148.9(3) |
| C(15)-Zr(1)-C(2) | 107.4(3) |
| C(5)-Zr(1)-C(2) | 137.8(3) |
| C(7)-Zr(1)-C(2) | 117.4(3) |
| C(9)-Zr(1)-C(2) | 106.1(3) |
| C(8)-Zr(1)-C(2) | 96.5(3) |
| C(42)-Zr(2)-C(59) | 95.8(3) |
| C(42)-Zr(2)-C(60) | 128.2(3) |
| C(59)-Zr(2)-C(60) | 33.6(3) |
| C(42)-Zr(2)-C(57) | 92.3(3) |
| C(59)-Zr(2)-C(57) | 54.8(3) |
| C(60)-Zr(2)-C(57) | 54.5(3) |
| C(42)-Zr(2)-C(58) | 76.1(3) |
| C(59)-Zr(2)-C(58) | 32.4(3) |
| C(60)-Zr(2)-C(58) | 54.2(3) |
| C(57)-Zr(2)-C(58) | 32.9(3) |
| C(42)-Zr(2)-C(44) | 58.3(3) |
| C(59)-Zr(2)-C(44) | 135.5(3) |
| C(60)-Zr(2)-C(44) | 140.1(3) |
| C(57)-Zr(2)-C(44) | 88.2(3) |
| C(58)-Zr(2)-C(44) | 103.1(3) |
| C(42)-Zr(2)-C(47) | 131.2(3) |
| C(59)-Zr(2)-C(47) | 99.7(3) |
| C(60)-Zr(2)-C(47) | 82.7(3) |
| C(57)-Zr(2)-C(47) | 133.8(3) |
| C(58)-Zr(2)-C(47) | 131.9(3) |
| C(44)-Zr(2)-C(47) | 124.7(3) |
| C(42)-Zr(2)-C(56) | 125.0(3) |
| C(59)-Zr(2)-C(56) | 54.9(3) |
| C(60)-Zr(2)-C(56) | 32.6(3) |
| C(57)-Zr(2)-C(56) | 32.9(3) |
| C(58)-Zr(2)-C(56) | 54.2(3) |
| C(44)-Zr(2)-C(56) | 108.0(3) |
| C(47)-Zr(2)-C(56) | 101.3(3) |
| C(42)-Zr(2)-C(50) | 78.5(3) |
| C(59)-Zr(2)-C(50) | 96.8(3) |
| C(60)-Zr(2)-C(50) | 109.4(3) |
| C(57)-Zr(2)-C(50) | 149.5(3) |
| C(58)-Zr(2)-C(50) | 116.9(3) |
| C(44)-Zr(2)-C(50) | 110.3(3) |
| C(47)-Zr(2)-C(50) | 54.0(3) |
| C(56)-Zr(2)-C(50) | 141.6(3) |
| C(42)-Zr(2)-C(46) | 107.9(3) |
| C(59)-Zr(2)-C(46) | 80.3(3) |
| C(60)-Zr(2)-C(46) | 80.2(3) |
| C(57)-Zr(2)-C(46) | 132.6(3) |
| C(58)-Zr(2)-C(46) | 110.6(3) |
| C(44)-Zr(2)-C(46) | 139.0(3) |
| C(47)-Zr(2)-C(46) | 32.7(3) |
| C(56)-Zr(2)-C(46) | 110.3(3) |
| C(50)-Zr(2)-C(46) | 32.6(3) |

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| C(59)-Zr(2)-C(48) | 131.7(3) |
| C(60)-Zr(2)-C(48) | 113.9(3) |
| C(57)-Zr(2)-C(48) | 152.8(3) |
| C(58)-Zr(2)-C(48) | 164.1(3) |
| C(44)-Zr(2)-C(48) | 92.6(3) |
| C(47)-Zr(2)-C(48) | 32.8(3) |
| C(56)-Zr(2)-C(48) | 123.1(3) |
| C(50)-Zr(2)-C(48) | 53.6(3) |
| C(46)-Zr(2)-C(48) | 54.1(3) |
| C(42)-Zr(2)-C(49) | 80.9(3) |
| C(59)-Zr(2)-C(49) | 128.6(3) |
| C(60)-Zr(2)-C(49) | 132.5(3) |
| C(57)-Zr(2)-C(49) | 172.6(3) |
| C(58)-Zr(2)-C(49) | 145.6(3) |
| C(44)-Zr(2)-C(49) | 85.6(3) |
| C(47)-Zr(2)-C(49) | 53.5(3) |
| C(56)-Zr(2)-C(49) | 154.0(3) |
| C(50)-Zr(2)-C(49) | 32.0(3) |
| C(46)-Zr(2)-C(49) | 53.4(3) |
| C(48)-Zr(2)-C(49) | 32.0(3) |
| C(42)-Zr(2)-C(43) | 32.3(3) |
| C(59)-Zr(2)-C(43) | 108.4(3) |
| C(60)-Zr(2)-C(43) | 129.6(3) |
| C(57)-Zr(2)-C(43) | 77.0(3) |
| C(58)-Zr(2)-C(43) | 78.1(3) |
| C(44)-Zr(2)-C(43) | 31.4(3) |
| C(47)-Zr(2)-C(43) | 147.8(3) |
| C(56)-Zr(2)-C(43) | 107.5(3) |
| C(50)-Zr(2)-C(43) | 106.0(3) |
| C(46)-Zr(2)-C(43) | 138.1(3) |
| C(48)-Zr(2)-C(43) | 115.8(3) |
| C(49)-Zr(2)-C(43) | 95.6(3) |
| C(2)-C(1)-Zr(1) | 80.9(5) |
| C(3)-C(2)-C(1) | 119.7(9) |
| C(3)-C(2)-C(4) | 119.2(8) |
| C(1)-C(2)-C(4) | 120.4(8) |
| C(3)-C(2)-Zr(1) | 71.1(5) |
| C(1)-C(2)-Zr(1) | 66.7(5) |
| C(4)-C(2)-Zr(1) | 127.5(6) |
| C(2)-C(3)-Zr(1) | 77.5(5) |
| C(9)-C(5)-C(6) | 108.7(7) |
| C(9)-C(5)-C(10) | 124.1(8) |
| C(6)-C(5)-C(10) | 126.1(8) |
| C(9)-C(5)-Zr(1) | 74.7(5) |
| C(6)-C(5)-Zr(1) | 72.9(5) |
| C(10)-C(5)-Zr(1) | 128.1(6) |
| C(7)-C(6)-C(5) | 107.2(8) |
| C(7)-C(6)-C(11) | 126.3(8) |
| C(5)-C(6)-C(11) | 124.7(8) |
| C(7)-C(6)-Zr(1) | 74.8(5) |
| C(5)-C(6)-Zr(1) | 74.3(5) |
| C(11)-C(6)-Zr(1) | 128.6(6) |
| C(6)-C(7)-C(8) | 108.4(7) |
| C(6)-C(7)-C(12) | 125.4(9) |
| C(8)-C(7)-C(12) | 125.9(8) |
| C(6)-C(7)-Zr(1) | 72.7(5) |
| C(8)-C(7)-Zr(1) | 75.1(5) |
| C(12)-C(7)-Zr(1) | 123.3(6) |
| C(7)-C(8)-C(9) | 108.5(8) |
| C(7)-C(8)-C(13) | 124.8(8) |
| C(9)-C(8)-C(13) | 126.0(9) |
| C(7)-C(8)-Zr(1) | 72.8(5) |
| C(9)-C(8)-Zr(1) | 73.3(5) |
| C(13)-C(8)-Zr(1) | 127.3(6) |
| C(5)-C(9)-C(8) | 107.2(8) |
| C(5)-C(9)-C(14) | 125.6(8) |
| C(8)-C(9)-C(14) | 126.7(8) |
| C(5)-C(9)-Zr(1) | 72.9(5) |

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| C(14)-C(9)-Zr(1) | 124.4(6) |
| C(19)-C(15)-C(16) | 107.1(7) |
| C(19)-C(15)-C(20) | 127.6(8) |
| C(16)-C(15)-C(20) | 125.3(8) |
| C(19)-C(15)-Zr(1) | 71.2(5) |
| C(16)-C(15)-Zr(1) | 73.1(5) |
| C(20)-C(15)-Zr(1) | 119.2(5) |
| C(15)-C(16)-C(17) | 108.0(8) |
| C(15)-C(16)-Zr(1) | 74.2(5) |
| C(17)-C(16)-Zr(1) | 73.3(5) |
| C(18)-C(17)-C(16) | 108.1(8) |
| C(18)-C(17)-Zr(1) | 71.4(5) |
| C(16)-C(17)-Zr(1) | 73.8(5) |
| C(19)-C(18)-C(17) | 107.6(8) |
| C(19)-C(18)-Zr(1) | 73.9(5) |
| C(17)-C(18)-Zr(1) | 75.6(5) |
| C(18)-C(19)-C(15) | 109.2(8) |
| C(18)-C(19)-Zr(1) | 72.9(5) |
| C(15)-C(19)-Zr(1) | 75.9(5) |
| C(15)-C(20)-C(22) | 107.3(7) |
| C(15)-C(20)-C(21) | 108.2(7) |
| C(22)-C(20)-C(21) | 107.5(8) |
| C(15)-C(20)-C(23) | 107.4(7) |
| C(22)-C(20)-C(23) | 111.8(7) |
| C(21)-C(20)-C(23) | 114.5(7) |
| C(20)-C(23)-B(1) | 120.9(7) |
| C(25)-C(24)-C(29) | 111.7(8) |
| C(25)-C(24)-B(1) | 120.2(7) |
| C(29)-C(24)-B(1) | 127.7(8) |
| C(26)-C(25)-F(1) | 115.1(8) |
| C(26)-C(25)-C(24) | 125.7(9) |
| F(1)-C(25)-C(24) | 119.1(7) |
| C(27)-C(26)-C(25) | 120.8(10) |
| C(27)-C(26)-F(2) | 118.6(9) |
| C(25)-C(26)-F(2) | 120.6(9) |
| F(3)-C(27)-C(26) | 122.8(10) |
| F(3)-C(27)-C(28) | 120.2(10) |
| C(26)-C(27)-C(28) | 116.9(9) |
| F(4)-C(28)-C(27) | 120.3(9) |
| F(4)-C(28)-C(29) | 118.7(10) |
| C(27)-C(28)-C(29) | 121.0(9) |
| F(5)-C(29)-C(28) | 115.7(8) |
| F(5)-C(29)-C(24) | 120.6(8) |
| C(28)-C(29)-C(24) | 123.6(9) |
| C(35)-C(30)-C(31) | 112.2(8) |
| C(35)-C(30)-B(1) | 126.8(7) |
| C(31)-C(30)-B(1) | 120.6(7) |
| C(32)-C(31)-F(6) | 115.6(7) |
| C(32)-C(31)-C(30) | 125.6(8) |
| F(6)-C(31)-C(30) | 118.9(7) |
| F(7)-C(32)-C(31) | 121.3(8) |
| F(7)-C(32)-C(33) | 119.2(8) |
| C(31)-C(32)-C(33) | 119.4(8) |
| F(8)-C(33)-C(34) | 121.1(8) |
| F(8)-C(33)-C(32) | 121.0(8) |
| C(34)-C(33)-C(32) | 117.9(8) |
| F(9)-C(34)-C(33) | 119.5(8) |
| F(9)-C(34)-C(35) | 119.6(8) |
| C(33)-C(34)-C(35) | 120.9(8) |
| F(10)-C(35)-C(34) | 114.5(8) |
| F(10)-C(35)-C(30) | 121.5(8) |
| C(34)-C(35)-C(30) | 123.9(8) |
| C(37)-C(36)-C(41) | 111.9(7) |
| C(37)-C(36)-B(1) | 128.1(7) |
| C(41)-C(36)-B(1) | 119.8(7) |
| F(11)-C(37)-C(38) | 113.6(7) |
| F(11)-C(37)-C(36) | 122.2(7) |
| C(38)-C(37)-C(36) | 124.2(8) |

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| F(12)-C(38)-C(37) | 120.6(8) |
| C(39)-C(38)-C(37) | 121.0(8) |
| F(13)-C(39)-C(38) | 121.9(8) |
| F(13)-C(39)-C(40) | 120.6(8) |
| C(38)-C(39)-C(40) | 117.5(8) |
| F(14)-C(40)-C(39) | 120.0(8) |
| F(14)-C(40)-C(41) | 120.0(8) |
| C(39)-C(40)-C(41) | 120.0(8) |
| F(15)-C(41)-C(40) | 115.8(7) |
| F(15)-C(41)-C(36) | 118.8(7) |
| C(40)-C(41)-C(36) | 125.3(8) |
| C(43)-C(42)-Zr(2) | 80.5(6) |
| C(44)-C(43)-C(42) | 119.3(9) |
| C(44)-C(43)-C(45) | 119.7(9) |
| C(42)-C(43)-C(45) | 120.5(9) |
| C(44)-C(43)-Zr(2) | 70.7(5) |
| C(42)-C(43)-Zr(2) | 67.3(5) |
| C(45)-C(43)-Zr(2) | 128.7(6) |
| C(43)-C(44)-Zr(2) | 77.9(6) |
| C(50)-C(46)-C(47) | 107.1(7) |
| C(50)-C(46)-C(51) | 126.6(8) |
| C(47)-C(46)-C(51) | 125.6(8) |
| C(50)-C(46)-Zr(2) | 73.1(5) |
| C(47)-C(46)-Zr(2) | 72.7(5) |
| C(51)-C(46)-Zr(2) | 127.0(6) |
| C(46)-C(47)-C(48) | 108.1(7) |
| C(46)-C(47)-C(52) | 125.6(8) |
| C(48)-C(47)-C(52) | 124.9(8) |
| C(46)-C(47)-Zr(2) | 74.6(5) |
| C(48)-C(47)-Zr(2) | 74.6(5) |
| C(52)-C(47)-Zr(2) | 127.7(5) |
| C(49)-C(48)-C(47) | 107.4(7) |
| C(49)-C(48)-C(53) | 126.4(8) |
| C(47)-C(48)-C(53) | 125.4(8) |
| C(49)-C(48)-Zr(2) | 74.9(5) |
| C(47)-C(48)-Zr(2) | 72.6(5) |
| C(53)-C(48)-Zr(2) | 126.2(6) |
| C(50)-C(49)-C(48) | 108.7(7) |
| C(50)-C(49)-C(54) | 125.7(8) |
| C(48)-C(49)-C(54) | 125.1(8) |
| C(50)-C(49)-Zr(2) | 72.4(4) |
| C(48)-C(49)-Zr(2) | 73.1(4) |
| C(54)-C(49)-Zr(2) | 127.0(6) |
| C(49)-C(50)-C(46) | 108.6(7) |
| C(49)-C(50)-C(55) | 127.8(8) |
| C(46)-C(50)-C(55) | 122.7(8) |
| C(49)-C(50)-Zr(2) | 75.6(5) |
| C(46)-C(50)-Zr(2) | 74.3(5) |
| C(55)-C(50)-Zr(2) | 124.9(6) |
| C(60)-C(56)-C(57) | 107.2(7) |
| C(60)-C(56)-C(61) | 126.6(7) |
| C(57)-C(56)-C(61) | 126.1(8) |
| C(60)-C(56)-Zr(2) | 71.7(4) |
| C(57)-C(56)-Zr(2) | 72.4(4) |
| C(61)-C(56)-Zr(2) | 120.1(5) |
| C(58)-C(57)-C(56) | 107.7(8) |
| C(58)-C(57)-Zr(2) | 74.0(5) |
| C(56)-C(57)-Zr(2) | 74.6(5) |
| C(59)-C(58)-C(57) | 108.8(8) |
| C(59)-C(58)-Zr(2) | 72.0(5) |
| C(57)-C(58)-Zr(2) | 73.1(5) |
| C(58)-C(59)-C(60) | 107.5(7) |
| C(58)-C(59)-Zr(2) | 75.5(5) |
| C(60)-C(59)-Zr(2) | 73.6(4) |
| C(56)-C(60)-C(59) | 108.7(7) |
| C(56)-C(60)-Zr(2) | 75.7(5) |
| C(59)-C(60)-Zr(2) | 72.8(5) |
| C(63)-C(61)-C(56) | 108.1(7) |

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| C(56)-C(61)-C(62) | 107.7(7) |
| C(63)-C(61)-C(64) | 112.5(7) |
| C(56)-C(61)-C(64) | 107.6(6) |
| C(62)-C(61)-C(64) | 112.2(6) |
| C(61)-C(64)-B(2) | 121.8(6) |
| C(66)-C(65)-C(70) | 112.9(7) |
| C(66)-C(65)-B(2) | 120.0(7) |
| C(70)-C(65)-B(2) | 126.8(7) |
| F(16)-C(66)-C(67) | 116.5(7) |
| F(16)-C(66)-C(65) | 119.2(7) |
| C(67)-C(66)-C(65) | 124.3(7) |
| F(17)-C(67)-C(68) | 119.7(8) |
| F(17)-C(67)-C(66) | 119.6(7) |
| C(68)-C(67)-C(66) | 120.7(8) |
| F(18)-C(68)-C(67) | 121.8(8) |
| F(18)-C(68)-C(69) | 120.6(8) |
| C(67)-C(68)-C(69) | 117.6(8) |
| F(19)-C(69)-C(70) | 120.4(8) |
| F(19)-C(69)-C(68) | 119.2(8) |
| C(70)-C(69)-C(68) | 120.4(8) |
| F(20)-C(70)-C(69) | 114.8(7) |
| F(20)-C(70)-C(65) | 121.0(7) |
| C(69)-C(70)-C(65) | 124.2(8) |
| C(76)-C(71)-C(72) | 112.9(8) |
| C(76)-C(71)-B(2) | 127.5(8) |
| C(72)-C(71)-B(2) | 119.4(7) |
| F(21)-C(72)-C(73) | 116.1(8) |
| F(21)-C(72)-C(71) | 120.0(8) |
| C(73)-C(72)-C(71) | 124.0(8) |
| F(22)-C(73)-C(74) | 119.2(8) |
| F(22)-C(73)-C(72) | 119.7(9) |
| C(74)-C(73)-C(72) | 121.0(8) |
| F(23)-C(74)-C(73) | 121.2(9) |
| F(23)-C(74)-C(75) | 121.1(10) |
| C(73)-C(74)-C(75) | 117.7(8) |
| F(24)-C(75)-C(74) | 120.4(9) |
| F(24)-C(75)-C(76) | 119.0(9) |
| C(74)-C(75)-C(76) | 120.5(9) |
| F(25)-C(76)-C(71) | 121.8(8) |
| F(25)-C(76)-C(75) | 114.4(7) |
| C(71)-C(76)-C(75) | 123.8(8) |
| C(82)-C(77)-C(78) | 112.1(7) |
| C(82)-C(77)-B(2) | 121.3(7) |
| C(78)-C(77)-B(2) | 126.2(7) |
| F(26)-C(78)-C(77) | 122.4(7) |
| F(26)-C(78)-C(79) | 113.1(8) |
| C(77)-C(78)-C(79) | 124.5(8) |
| F(27)-C(79)-C(80) | 120.3(8) |
| F(27)-C(79)-C(78) | 119.9(9) |
| C(80)-C(79)-C(78) | 119.8(8) |
| F(28)-C(80)-C(81) | 122.0(9) |
| F(28)-C(80)-C(79) | 119.8(9) |
| C(81)-C(80)-C(79) | 118.2(8) |
| F(29)-C(81)-C(80) | 119.4(8) |
| F(29)-C(81)-C(82) | 121.3(8) |
| C(80)-C(81)-C(82) | 119.4(8) |
| F(30)-C(82)-C(77) | 119.6(7) |
| F(30)-C(82)-C(81) | 114.4(8) |
| C(77)-C(82)-C(81) | 126.0(8) |
| Cl(2)-C(83)-Cl(1) | 113.3(9) |
| Cl(3)-C(84)-Cl(4) | 116.6(13) |
| Cl(5)-C(85)-Cl(6) | 115.5(8) |
| C(24)-B(1)-C(23) | 105.8(7) |
| C(24)-B(1)-C(36) | 108.6(7) |
| C(23)-B(1)-C(36) | 112.0(7) |
| C(24)-B(1)-C(30) | 113.0(7) |
| C(23)-B(1)-C(30) | 116.4(7) |
| C(36)-B(1)-C(30) | 101.0(6) |

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| C(65)-B(2)-C(71) | 113.[illegible](7) |
| C(65)-B(2)-C(77) | 110.4(6) |
| C(71)-B(2)-C(77) | 100.3(6) |
| C(65)-B(2)-C(64) | 105.3(6) |
| C(71)-B(2)-C(64) | 115.2(7) |
| C(77)-B(2)-C(64) | 112.1(6) |

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Zr(1) | 26(1) | 27(1) | 22(1) | 8(1) | 1(1) | 5(1) |
| Zr(2) | 23(1) | 28(1) | 23(1) | 10(1) | -1(1) | 2(1) |
| Cl(1) | 73(2) | 156(4) | 69(2) | 8(2) | 5(2) | 34(3) |
| Cl(2) | 90(3) | 193(5) | 173(5) | 145(4) | 0(3) | -6(3) |
| Cl(3) | 114(4) | 99(4) | 380(11) | 62(5) | -92(5) | 6(3) |
| Cl(4) | 90(4) | 184(7) | 511(16) | 112(9) | -49(6) | -59(4) |
| Cl(5) | 208(6) | 113(4) | 113(4) | 10(3) | 45(4) | 29(4) |
| Cl(6) | 169(5) | 173(5) | 88(3) | 5(3) | 47(3) | -61(4) |
| F(1) | 30(3) | 39(3) | 40(3) | 7(2) | 6(2) | 1(2) |
| F(2) | 71(4) | 56(4) | 60(4) | 14(3) | 20(3) | -17(3) |
| F(3) | 129(6) | 29(3) | 70(4) | 20(3) | 25(4) | 0(4) |
| F(4) | 103(5) | 34(3) | 50(4) | 16(3) | 6(3) | 28(3) |
| F(5) | 43(3) | 39(3) | 34(3) | 9(2) | 6(2) | 19(3) |
| F(6) | 43(3) | 43(3) | 20(3) | 0(2) | 0(2) | 1(3) |
| F(7) | 54(4) | 81(4) | 23(3) | 4(3) | 15(3) | 1(3) |
| F(8) | 47(4) | 74(4) | 45(3) | 19(3) | 15(3) | -6(3) |
| F(9) | 34(3) | 47(3) | 56(4) | 13(3) | -1(3) | -5(3) |
| F(10) | 35(3) | 35(3) | 31(3) | -5(2) | -3(2) | 5(2) |
| F(11) | 45(3) | 31(3) | 27(3) | 3(2) | -9(2) | 2(2) |
| F(12) | 50(4) | 53(4) | 42(3) | 8(3) | -22(3) | 6(3) |
| F(13) | 58(4) | 64(4) | 53(4) | 23(3) | -18(3) | 27(3) |
| F(14) | 57(4) | 37(3) | 46(3) | 19(3) | -5(3) | 11(3) |
| F(15) | 47(3) | 26(3) | 34(3) | 10(2) | -10(2) | -3(2) |
| F(16) | 42(3) | 26(3) | 33(3) | 16(2) | -4(2) | 4(2) |
| F(17) | 49(3) | 29(3) | 52(3) | 14(3) | -4(3) | -6(3) |
| F(18) | 40(3) | 44(3) | 55(4) | 9(3) | -13(3) | -10(3) |
| F(19) | 35(3) | 56(4) | 37(3) | 17(3) | -15(2) | 4(3) |
| F(20) | 36(3) | 29(3) | 30(3) | 14(2) | 1(2) | 7(2) |
| F(21) | 36(3) | 38(3) | 42(3) | 10(2) | 8(2) | 10(2) |
| F(22) | 61(4) | 42(3) | 59(4) | 13(3) | 11(3) | 25(3) |
| F(23) | 96(5) | 22(3) | 71(4) | 13(3) | -1(4) | 8(3) |
| F(24) | 59(4) | 38(3) | 52(4) | 17(3) | 0(3) | -13(3) |
| F(25) | 30(3) | 42(3) | 41(3) | 17(2) | 0(2) | -5(2) |
| F(26) | 33(3) | 58(4) | 31(3) | 4(3) | 13(2) | -8(3) |
| F(27) | 66(4) | 60(4) | 37(3) | 14(3) | 29(3) | 4(3) |
| F(28) | 77(4) | 72(4) | 17(3) | 7(3) | 8(3) | 15(3) |
| F(29) | 61(4) | 64(4) | 26(3) | -5(3) | -8(3) | 9(3) |
| F(30) | 34(3) | 37(3) | 28(3) | -1(2) | 0(2) | -2(2) |
| C(1) | 36(5) | 39(6) | 22(5) | 6(4) | 6(4) | 6(5) |
| C(2) | 36(5) | 23(5) | 35(5) | 8(4) | 1(4) | -6(4) |
| C(3) | 35(6) | 35(6) | 30(5) | 14(4) | 6(4) | 5(4) |
| C(4) | 38(6) | 43(6) | 48(6) | 9(5) | 1(5) | -11(5) |
| C(5) | 28(5) | 41(6) | 31(5) | 18(4) | 14(4) | 15(4) |
| C(6) | 22(5) | 38(5) | 36(5) | 13(4) | -3(4) | 3(4) |
| C(7) | 19(5) | 55(6) | 25(5) | 23(5) | 6(4) | 12(4) |
| C(8) | 25(5) | 42(6) | 41(6) | 22(5) | 5(4) | 12(4) |
| C(9) | 27(5) | 35(5) | 29(5) | 6(4) | 6(4) | 11(4) |
| C(10) | 40(6) | 65(7) | 39(6) | 26(5) | 12(5) | 16(5) |
| C(11) | 27(5) | 47(6) | 55(6) | 20(5) | 5(5) | -4(5) |
| C(12) | 44(6) | 81(8) | 30(5) | 20(5) | 5(5) | 9(6) |
| C(13) | 41(6) | 60(7) | 70(7) | 43(6) | 9(5) | 22(5) |
| C(14) | 49(7) | 47(6) | 51(7) | 3(5) | 11(5) | 20(5) |
| C(15) | 32(5) | 29(5) | 21(4) | 7(4) | -5(4) | 13(4) |
| C(16) | 27(5) | 31(5) | 22(5) | 8(4) | 2(4) | 10(4) |
| C(17) | 34(5) | 49(6) | 28(5) | 15(5) | -4(4) | 6(5) |
| C(18) | 30(5) | 42(6) | 25(5) | 12(4) | 1(4) | 10(4) |
| C(19) | 24(5) | 38(5) | 25(5) | 17(4) | -2(4) | 10(4) |
| C(20) | 37(5) | 32(5) | 21(5) | 5(4) | -2(4) | 8(4) |
| C(21) | 53(6) | 47(6) | 26(5) | 7(4) | 7(4) | 30(5) |
| C(22) | 69(7) | 29(5) | 27(5) | 13(4) | -7(5) | 1(5) |
| C(23) | 27(5) | 26(5) | 15(4) | 8(4) | -4(3) | 2(4) |
| C(24) | 29(5) | 28(5) | 15(4) | 6(4) | -1(4) | 3(4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(26) | 58(7) | 35(6) | 31(5) | 4(4) | 4(5) | -9(5) |
| C(27) | 81(8) | 29(6) | 36(6) | 15(5) | 11(6) | -3(6) |
| C(28) | 85(8) | 28(6) | 25(5) | 11(4) | 4(5) | 25(6) |
| C(29) | 40(6) | 32(5) | 16(4) | 7(4) | 8(4) | 21(4) |
| C(30) | 30(5) | 28(5) | 21(5) | 4(4) | -7(4) | 6(4) |
| C(31) | 33(5) | 31(5) | 23(5) | 7(4) | -2(4) | -2(4) |
| C(32) | 39(6) | 46(6) | 27(5) | 12(5) | 5(4) | 1(5) |
| C(33) | 30(5) | 51(6) | 34(5) | 18(5) | 9(4) | 0(5) |
| C(34) | 36(6) | 37(6) | 32(5) | 6(4) | -7(4) | -2(5) |
| C(35) | 34(5) | 32(5) | 22(5) | 6(4) | -3(4) | 11(4) |
| C(36) | 21(4) | 33(5) | 17(4) | 10(4) | 4(3) | 7(4) |
| C(37) | 32(5) | 24(5) | 23(5) | 8(4) | -4(4) | 0(4) |
| C(38) | 29(5) | 42(6) | 29(5) | 8(4) | -8(4) | -1(4) |
| C(39) | 36(5) | 41(6) | 30(5) | 12(4) | -1(4) | 17(5) |
| C(40) | 45(6) | 18(5) | 33(5) | 11(4) | 6(4) | 5(4) |
| C(41) | 27(5) | 35(5) | 19(4) | 9(4) | 1(4) | 5(4) |
| C(42) | 30(6) | 49(6) | 43(6) | 17(5) | -4(5) | -5(5) |
| C(43) | 33(5) | 35(5) | 30(5) | 10(4) | -2(4) | -6(4) |
| C(44) | 31(5) | 26(5) | 38(6) | 6(4) | 0(5) | -6(4) |
| C(45) | 51(7) | 51(7) | 63(7) | 36(6) | 5(6) | -7(5) |
| C(46) | 30(5) | 31(5) | 22(5) | 5(4) | 1(4) | 6(4) |
| C(47) | 27(5) | 27(5) | 30(5) | 14(4) | -2(4) | 1(4) |
| C(48) | 36(5) | 30(5) | 24(5) | 16(4) | 6(4) | 6(4) |
| C(49) | 39(5) | 28(5) | 20(5) | 13(4) | -12(4) | -5(4) |
| C(50) | 25(5) | 33(5) | 26(5) | 13(4) | 1(4) | 5(4) |
| C(51) | 59(7) | 35(6) | 33(5) | 13(4) | -2(5) | 10(5) |
| C(52) | 33(5) | 32(5) | 46(6) | 20(5) | -3(4) | -5(4) |
| C(53) | 46(6) | 37(6) | 41(6) | 25(5) | 19(5) | 20(5) |
| C(54) | 65(7) | 38(6) | 28(5) | 3(4) | -13(5) | -8(5) |
| C(55) | 24(5) | 56(7) | 60(7) | 30(6) | 9(5) | 5(5) |
| C(56) | 26(5) | 32(5) | 23(5) | 13(4) | -1(4) | 3(4) |
| C(57) | 29(5) | 38(5) | 20(4) | 12(4) | -5(4) | -1(4) |
| C(58) | 23(5) | 63(7) | 29(5) | 26(5) | 12(4) | 12(5) |
| C(59) | 29(5) | 43(6) | 19(4) | 7(4) | 5(4) | 22(4) |
| C(60) | 26(5) | 28(5) | 20(4) | 7(4) | 2(4) | 5(4) |
| C(61) | 28(5) | 30(5) | 17(4) | 7(4) | 1(4) | 5(4) |
| C(62) | 29(5) | 36(5) | 16(4) | 2(4) | 5(4) | 11(4) |
| C(63) | 26(5) | 46(6) | 24(5) | 13(4) | 5(4) | 9(4) |
| C(64) | 20(4) | 24(4) | 24(4) | 12(4) | -3(3) | 5(4) |
| C(65) | 23(4) | 28(5) | 16(4) | 3(4) | 2(3) | 7(4) |
| C(66) | 26(5) | 32(5) | 18(4) | 4(4) | 3(4) | 7(4) |
| C(67) | 29(5) | 28(5) | 31(5) | 14(4) | 11(4) | 5(4) |
| C(68) | 28(5) | 31(5) | 30(5) | 1(4) | 4(4) | 1(4) |
| C(69) | 27(5) | 34(5) | 23(5) | 6(4) | 4(4) | 7(4) |
| C(70) | 24(5) | 29(5) | 25(5) | 8(4) | 2(4) | 1(4) |
| C(71) | 34(5) | 27(5) | 19(4) | 8(4) | -3(4) | -1(4) |
| C(72) | 33(5) | 31(5) | 25(5) | 6(4) | -1(4) | -1(4) |
| C(73) | 38(6) | 37(6) | 35(5) | 7(4) | 3(4) | 20(5) |
| C(74) | 68(8) | 26(5) | 34(6) | 8(4) | -6(5) | 10(5) |
| C(75) | 48(6) | 36(6) | 27(5) | 13(4) | -2(4) | -5(5) |
| C(76) | 28(5) | 32(5) | 24(5) | 10(4) | -2(4) | -1(4) |
| C(77) | 35(5) | 20(4) | 23(5) | 4(4) | 6(4) | 4(4) |
| C(78) | 38(5) | 21(5) | 25(5) | -1(4) | -4(4) | 2(4) |
| C(79) | 54(6) | 36(5) | 30(5) | 15(4) | 25(5) | 7(5) |
| C(80) | 54(7) | 37(6) | 25(5) | 6(4) | 2(5) | 11(5) |
| C(81) | 44(6) | 35(5) | 25(5) | 3(4) | -5(4) | 7(5) |
| C(82) | 32(5) | 26(5) | 22(5) | 4(4) | 6(4) | 5(4) |
| C(83) | 81(10) | 96(11) | 68(9) | 22(8) | 13(7) | 28(8) |
| C(84) | 103(16) | 172(22) | 363(38) | 158(25) | -92(20) | -65(15) |
| C(85) | 174(18) | 121(13) | 89(12) | 50(10) | 97(12) | 92(13) |
| B(1) | 31(6) | 29(6) | 24(5) | 10(4) | 1(4) | 9(5) |
| B(2) | 18(5) | 25(5) | 27(5) | 10(4) | 2(4) | 5(4) |

Table 5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2x10^3$) for 1.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 2999(74) | 10549(51) | 6743(44) | 39 |
| H(1B) | 4033(71) | 10548(51) | 7270(49) | 39 |
| H(3A) | 4100(75) | 9471(53) | 5264(48) | 39 |
| H(3B) | 2964(75) | 9796(50) | 5490(44) | 39 |
| H(4A) | 5727(8) | 10010(37) | 6751(15) | 67 |
| H(4B) | 5830(7) | 10573(15) | 6293(34) | 67 |
| H(4C) | 5746(9) | 9669(24) | 5937(20) | 67 |
| H(10A) | 1058(51) | 8171(13) | 7553(12) | 67 |
| H(10B) | 177(16) | 8761(36) | 7748(20) | 67 |
| H(10C) | 1403(37) | 9035(25) | 8012(9) | 67 |
| H(11A) | -340(7) | 7554(6) | 6031(32) | 63 |
| H(11B) | 676(41) | 7366(11) | 6427(16) | 63 |
| H(11C) | 700(41) | 7311(9) | 5634(16) | 63 |
| H(12A) | 1090(40) | 8947(26) | 4845(6) | 76 |
| H(12B) | -107(16) | 8683(38) | 4967(5) | 76 |
| H(12C) | 785(53) | 8099(13) | 4874(7) | 76 |
| H(13A) | 1548(45) | 10827(12) | 6316(20) | 76 |
| H(13B) | 365(11) | 10506(25) | 5979(34) | 76 |
| H(13C) | 1371(52) | 10274(15) | 5543(15) | 76 |
| H(14A) | 1759(50) | 10856(11) | 7476(10) | 76 |
| H(14B) | 1879(46) | 10327(15) | 7961(14) | 76 |
| H(14C) | 725(10) | 10541(24) | 7769(22) | 76 |
| H(16A) | 5172(7) | 8530(5) | 6220(4) | 32 |
| H(17A) | 4876(7) | 9362(6) | 7429(4) | 44 |
| H(18A) | 3176(7) | 8796(5) | 7798(4) | 38 |
| H(19A) | 2506(7) | 7572(5) | 6860(4) | 33 |
| H(21A) | 4822(10) | 6518(26) | 4980(15) | 62 |
| H(21B) | 5170(21) | 6705(34) | 5779(18) | 62 |
| H(21C) | 5320(15) | 7360(9) | 5418(31) | 62 |
| H(22A) | 3090(42) | 5973(9) | 5338(15) | 62 |
| H(22B) | 2312(14) | 6526(15) | 5793(31) | 62 |
| H(22C) | 3369(31) | 6337(22) | 6147(18) | 62 |
| H(23A) | 3357(7) | 7971(5) | 5132(4) | 27 |
| H(23B) | 2323(7) | 7422(5) | 5147(4) | 27 |
| H(42A) | 9787(75) | 7215(54) | 9793(51) | 49 |
| H(42B) | 10043(83) | 7323(60) | 9101(53) | 49 |
| H(44A) | 7499(75) | 8265(54) | 9870(47) | 39 |
| H(44B) | 8019(71) | 7711(52) | 10187(47) | 39 |
| H(45A) | 9248(54) | 8243(12) | 8572(14) | 78 |
| H(45B) | 9534(42) | 8938(21) | 9262(18) | 78 |
| H(45C) | 8317(15) | 8695(31) | 8961(31) | 78 |
| H(51A) | 7738(23) | 4384(21) | 8286(17) | 62 |
| H(51B) | 8450(50) | 4105(11) | 8814(7) | 62 |
| H(51C) | 8998(29) | 4631(12) | 8404(21) | 62 |
| H(52A) | 6037(21) | 4508(20) | 9430(24) | 53 |
| H(52B) | 6024(21) | 4704(27) | 8721(6) | 53 |
| H(52C) | 5485(7) | 5234(9) | 9360(26) | 53 |
| H(53A) | 6409(36) | 6033(21) | 10890(21) | 56 |
| H(53B) | 5825(14) | 6351(32) | 10338(5) | 56 |
| H(53C) | 6746(23) | 6883(13) | 10876(21) | 56 |
| H(54A) | 9182(48) | 6557(10) | 11201(13) | 70 |
| H(54B) | 8606(24) | 7238(21) | 11052(19) | 70 |
| H(54C) | 9735(25) | 7040(29) | 10757(8) | 70 |
| H(55A) | 10447(13) | 5966(32) | 9260(25) | 66 |
| H(55B) | 10320(9) | 5173(6) | 9430(31) | 66 |
| H(55C) | 10584(9) | 5959(32) | 10037(9) | 66 |
| H(57A) | 7344(7) | 7544(5) | 8155(4) | 34 |
| H(58A) | 9075(7) | 6920(6) | 7899(4) | 42 |
| H(59A) | 8717(7) | 5526(5) | 7810(4) | 36 |
| H(60A) | 6710(6) | 5247(5) | 7903(4) | 29 |
| H(62A) | 4820(34) | 5396(11) | 8065(14) | 42 |
| H(62B) | 4955(29) | 5497(14) | 7325(12) | 42 |

| | | | | |
|---|---|---|---|---|
| H(63A) | 5473(31) | 7582(11) | 7920(19) | 46 |
| H(63B) | 4283(13) | 7163(25) | 7776(24) | 46 |
| H(63C) | 5159(42) | 6875(15) | 7243(6) | 46 |
| H(64A) | 5847(6) | 7229(5) | 9137(4) | 26 |
| H(64B) | 5286(6) | 6420(5) | 9120(4) | 26 |
| H(83A) | 2107(11) | 478(8) | 3200(7) | 98 |
| H(83B) | 1051(11) | 906(8) | 3350(7) | 98 |
| H(84A) | 8417(16) | 6217(13) | 4659(15) | 245 |
| H(84B) | 8962(16) | 5879(13) | 3960(15) | 245 |
| H(85A) | 7557(15) | 246(10) | -1083(8) | 140 |
| H(85B) | 7755(15) | 1155(10) | -865(8) | 140 |